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R. F. Rakowski



## DEVELOPMENT OF A COLORIMETRIC PERSONAL DOSIMETER FOR NITROGEN DIOXIDE

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## **Foreword**

This report was prepared by members of the Chemical Hazards Branch, Toxic Hazards Division, Aerospace Medical Research Laboratory, Wright-Patterson Air Force Base, Ohio. The work was performed in support of project 6302, "Toxic Hazards of Propellants and Materials," task 630203, "Identification of Toxic Materials." The report covers work conducted from July 1967 to May 1968.

This technical report has been reviewed and is approved.

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## **Abstract**

A personal colorimetric dosimeter for nitrogen dioxide has been developed. Detector strips are prepared by dipping Eastman Chromagram Sheet, Type K301R2, in a solution of diphenylamine and oxalic acid in methanol. When dry, the strips can be used to estimate the concentration-time product to which an individual has been exposed. The strips are sensitive to heat and light. Nitrogen dioxide develops a green color while other oxidizers develop a blue color. The strips are not affected by moisture and should have a shelf life greater than one year. Color standards for concentration-time products of 50, 100, and 300 ppm-minutes and a blue standard to indicate an expired strip are described. A plastic holder for the sensitive detector strips is shown.

## **Section I.**

### **INTRODUCTION**

An investigation was conducted to develop a personal colorimetric dosimeter for nitrogen dioxide. Nitrogen tetroxide and red fuming nitric acid have been used as oxidizers in operational missile systems. It is desirable to have a simple, inexpensive device capable of warning a person of a hazardous concentration of NO<sub>2</sub> vapors and giving an estimate of the extent of the hazard.

The use of reagents impregnated on filter paper for the detection of various chemicals is widely known. However, these detectors have not been used to obtain quantitative information chiefly because of the great inaccuracy of the method.

A dosimeter which is capable of giving a quantitative estimate of the concentration-time product of an exposure to hydrazine fuels was developed by the Mine Safety Appliances Company under contract to the Air Force (ref 12). An evaluation of its performance has been made (ref 2).

The requirements for a satisfactory dosimeter for a hazardous chemical are the following:

1. Color development on the detector strip should take place rapidly, be readily observable and reasonably permanent.
2. The device must be simple to use and inexpensive to manufacture. It should not require any manual operation by the user other than visual inspection.
3. The color reaction should not be subject to excessive interference from other chemicals, moisture, heat, or light.
4. The sensitivity of the badge and the range of the color standards should correspond to the concentration range of toxicological interest.
5. The accuracy of the quantitative estimate of concentration-time product need not be very high but should be capable of indicating safe, borderline and unsafe conditions.
6. The sensitive material should have a shelf life longer than one year.

A survey of the literature was conducted to select reagents as candidates for a nitrogen dioxide dosimeter. This report describes the tests which were conducted on the most promising reagents and the development of a prototype dosimeter using diphenylamine and oxalic acid on a silica gel support.

## Section II.

### CANDIDATE COLOR REACTIONS FOR NITROGEN DIOXIDE

The Saltzman method is probably the most widely used technique for the colorimetric determination of low concentrations of NO<sub>2</sub> in air (ref 13, 14). A combination of N-(1-naphthyl)-ethylenediamine dihydrochloride and sulfanilic acid is used in an acetic acid solution. Kinoshian and Hubbard have described a squeeze bulb indicator tube for NO<sub>2</sub> which uses Saltzman reagent on silica gel (ref 6).

The Griess-Ilosvay reagent consisting of alpha-naphthylamine and sulfanilic acid in an acid solution is still used to some extent. Acetic acid is used in wet determinations (ref 18) while succinic, oxalic or tartaric acids can be used to prepare a stable dry powder that can be dissolved just prior to use (ref 16).

Various derivatives of diphenylamine and benzidine have been used for the determination of NO<sub>2</sub>. The oxidation of these reagents in the presence of strong acid gives blue colored pigments. Kitigawa described a detector tube using o-tolidine chloride on silica gel (ref 5) and Plantz described the use of tetraphenylbenzidine (ref 11). One detector tube for NO<sub>2</sub> uses carbazole and a second type uses diphenylbenzidine as a reagent. Diphenylamine will give a blue color with NO<sub>2</sub> in strong acid and has been used for a spot plate technique to determine nitrates (ref 10). In a neutral medium, the yellow nitro derivatives of diphenylamine will be formed. An early paper described the use of a moist strip of filter paper soaked in an alcoholic solution of diphenylamine to detect NO<sub>2</sub> formed by sparks (ref 17).

It is also possible to use a wide variety of acid-base or oxidation-reduction indicators as a means of detecting and estimating NO<sub>2</sub> concentrations. Starch iodide paper and litmus paper have been used with some success (ref 3). These tests, of course, are not specific. Aromatic nitro compounds are usually colored and a wide range of aromatic compounds have been used although they are usually not very sensitive.

Two classes of reactions appeared to be the most promising to investigate: diazotization reactions such as the Saltzman and Griess reactions and the reactions of various diphenylamine and benzidine derivatives.

## Section III.

### APPARATUS

The apparatus used to test the detector strips is shown in figure 1. Liquid nitrogen tetroxide is placed in a 250-ml, round-bottom flask having a 24/40 standard taper neck and a glass thermometer well. The flask containing  $N_2O_4$  is immersed in an ice bath at  $0^\circ C$  in a 4-liter Dewar flask. Diffusion tubes of various dimensions were fabricated from glass tubing and glass male joints and were inserted in the neck of the round bottom flask. Ambient air is drawn into a gas-mixing vessel fabricated from a 100-ml, round-bottom flask and 7-mm diameter glass tubing, where it is mixed with the small quantity of  $N_2O_4/NO_2$  vapor which has diffused up the tube. The mixture is drawn into a 250-ml, three-necked flask used as a testing chamber. A short, gum rubber connecting hose is used to link the diffusion cell to a gas inlet fabricated from glass tubing and a male glass joint. Another short, gum rubber connector is used to link the gas outlet to the remainder of the system.

The concentration in the test chamber is controlled by using different diffusion tubes or by changing the dilution rate. The system requires about 2 hours to equilibrate after initial assembly. The equilibrium concentration can be conveniently measured by withdrawing 40-ml samples into a glass syringe using a needle inserted into the rubber connectors. The Saltzman colorimetric technique was used to determine the concentration of  $NO_2$  in the test chamber (ref 13). Successive determinations of the concentration performed on a Bausch and Lomb Spectronic 20 Spectrophotometer indicated that the equilibrium concentration could be maintained to within about

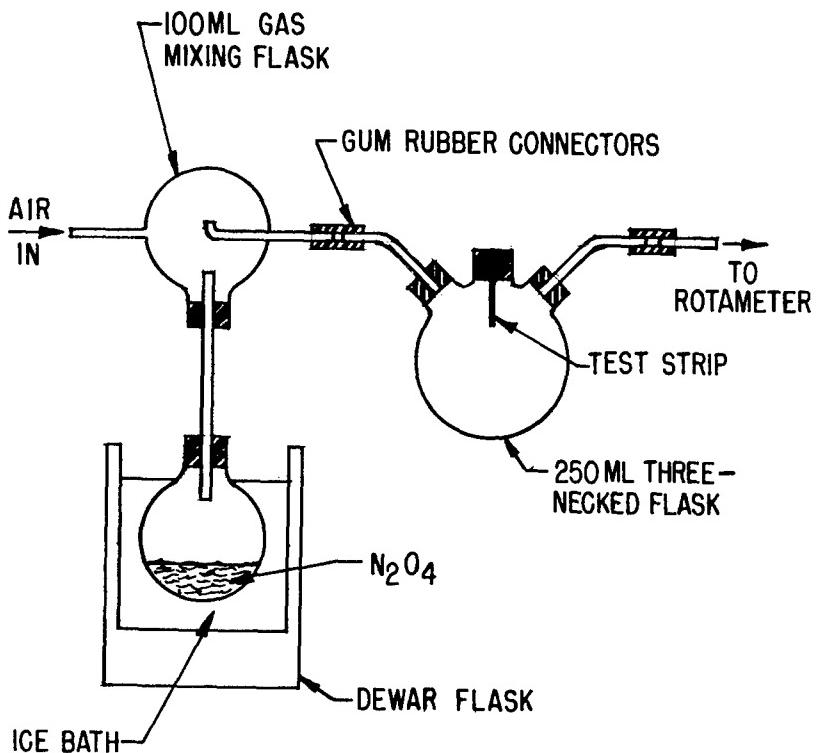


Figure 1. Apparatus used to test the detector strips

5 ppm at a concentration of 40 ppm. The concentration of NO<sub>2</sub> that results at an air flow rate of 4.5 liters/minute is given in table I. This method of obtaining low concentrations of volatile liquids was described by McKelvey and Hoelscher (ref 9). It is believed that this is the first reported use of their diffusion cell for producing low NO<sub>2</sub> concentrations.

The detector strip to be tested was inserted in a slit cut in a rubber stopper. Between runs a glass standard taper stopper was placed in the middle neck of the three-necked test chamber. The glass stopper was rapidly withdrawn and the rubber stopper holding the test strip inserted in the gas stream. At the end of a timed interval the test strip was withdrawn and the glass stopper replaced. A Mine Safety Appliances "Billion-Aire®" trace gas analyzer was used to monitor the concentration during the run. The Billion-Aire settings were adjusted so that the indicating meter was at mid-scale. A Dynatronic "Graphicorder" was used to record the meter reading for a few early runs. The graphical output indicated that the upset caused by the insertion of the test strips was less than 10 seconds in duration and that the system rapidly returned to the equilibrium concentration during the run. Concentration-time products for runs of one minute can be expected to have the largest percent error. Runs were not conducted for time periods less than one minute. A few runs were made that were 30 minutes long.

The gas flow rate was monitored by an all glass rotameter with a tantalum float that had been calibrated with air. Suction was provided by the diaphragm pump of the Billion-Aire. The entire apparatus was assembled in a ventilated hood. The vent line from the Billion-Aire was placed directly in an exhaust duct.

TABLE I.  
NO<sub>2</sub> CONCENTRATION FOR VARIOUS DIFFUSION TUBES

<i>Tube</i>	<i>Length (cm)</i>	<i>I.D. (cm)</i>	<i>Concentration of NO<sub>2</sub> (ppm)</i>
1	20	0.4	10 ± 1
2	20	0.7	25 ± 3
3	10	0.6	40 ± 5

Air dilution rate 4.5 liters/min  
Bath temperature 0°C

## Section IV.

### TEST OF SALTZMAN AND GREISS REAGENTS

A solution of 10 grams of sulfanilic acid and 0.8 gram of N-(1-naphthyl)-ethylenediamine dihydrochloride in one liter of water was prepared. Strips of Eastman Chromagram Sheet and filter paper were dipped in the solution and dried in air. Eastman Chromagram Sheet (Type K301R2) consists of a uniform layer of silica gel on polyethylene terephthalate with a polyvinyl alcohol binder. Freshly prepared strips that were still slightly moist showed excellent sensitivity to NO<sub>2</sub>. However, the sensitivity decreased markedly as the strips were dried, and the pink color did not develop at all on strips which were dried in a vacuum dessicator. To increase the amount of moisture present on the strip, various formulations were tried, including an efflorescent salt and a deliquescent salt. However, the sensitivity of these strips did not remain constant after storage. Similar tests were conducted with a combination of alpha-naphthylamine and sulfanilic acid, without success. The silica gel indicator tube developed by Kinosian and Hubbard using Saltzman's reagents depends on the length of stain developed in a sample tube rather than a color comparison and is less affected by small changes in moisture content of the gel. The NO<sub>2</sub> is adsorbed on the silica gel and the reagent only serves to indicate the depth to which the NO<sub>2</sub> has penetrated. The absorbing capacity of the silica gel determines the sensitivity of the indicator tube. It might be possible to develop a detector strip using a high moisture content gel, or a strip which is moistened just prior to use. However, a dry strip seems preferable, and tests with dry diphenylamine strips gave excellent results.

## Section V.

### PREPARATION AND TESTING OF DIPHENYLAMINE DETECTOR STRIPS

Diphenylamine and oxalic acid in a methanol solution was selected as the best choice among the various reagent combinations that were tested. Detector strips prepared from these reagents develop a forest green color when exposed to NO<sub>2</sub>, while reaction with other oxidizers produces a blue color.

A 5 x 20 cm strip of Eastman Chromagram Sheet, Type K301R2, was dipped briefly in a solution of diphenylamine and 1.25 grams of oxalic acid trihydrate in 100 ml of anhydrous methanol until completely wetted, allowed to drain and then dried in ambient air for about 15 minutes. The air dried strips were placed in a calcium chloride dessicator until packaged. The 5 x 20 cm sheets were cut into twenty 1 x 5 cm strips and placed in 3 dram screw cap, glass vials. A large quantity of detector strips were prepared and packaged for testing and evaluation.

The results of one series of tests that were run at 40 ppm of NO<sub>2</sub> using diffusion tube #3 are shown in table II. The colors are recorded by comparison with the standard colors given in Maerz and Paul's "Dictionary of Color" (ref 8).

A series of color standards was prepared by painting filter paper with tempera paints that had been mixed carefully to give colors that would match the Maerz and Paul standards. The filter paper was cut into small 1 x 1½ cm rectangles and used as permanent color standards fastened to each dosimeter badge.

TABLE II.  
RESPONSE OF DETECTOR STRIPS TO 40 PPM NO<sub>2</sub>

<i>CT Value (ppm-minutes)</i>	<i>Maerz and Paul Color Standard (Ref 8)</i>	<i>Badge Standard</i>
40	17E2	A
40	17E2	A
100	17G3	B
100	17G4	B
200	19H5	C
200	19H4	C
300	20J3	C
300	20J4	C

TABLE III.  
SELECTION OF COLOR STANDARDS

<i>CT Value ppm-min</i>	<i>Standard</i>	<i>Maers &amp; Paul (ref 8) Color Standard</i>	<i>Color</i>
Expired	X	33B1	light blue
0	O	17A1	white
50	A	17E2	light green-yellow
100	B	17I4	light green
300	C	19I5	green

The colors that were prepared as standards are given in table III. Since the detector strips become light blue when exposed to bright sunlight or other oxidizers, a standard was prepared that would indicate that the detector strip could not be used without incurring an error of greater than one color standard. The light-blue "X" standard represents the color that can be expected to develop indoors in about 8 hours or in direct sunlight in about 2 hours. If the strip becomes a darker blue, it should be discarded and a new strip put in place.

The blue "X" standard can also be used to determine if old strips are still usable. If the glass vials are not properly sealed during storage or the strips are exposed to excessive heat, the strips turn blue and should not be used.

The "O" standard is white and indicates the color of an unexposed strip. Although this standard is not necessary it makes the comparison of badges exposed to low NO<sub>2</sub> concentrations somewhat easier.

The "A," "B" and "C" standards are used to estimate the concentration-time product to which the badge has been exposed. These standards were selected after many tests as the most representative of colors produced under various conditions. The exact hue that a badge develops is dependent on the proportions of yellow and blue pigment present. A strip that is near the "X" standard when exposed to NO<sub>2</sub> will appear more blue-green than a strip that was initially white. Despite this variation in hue, it is fairly simple to match the appropriate standard by judging the darkness of the color and not its hue. A strip would be called "B" if it were judged to be darker than "A" but not as dark as "C." Since "A," "B" and "C" standards differ by a considerable amount, this judgment is not difficult. The color should be compared to the standard immediately after the exposure. The color is fairly stable, but slowly becomes yellow in about 10 hours.

In order to judge the effect of air oxidation, several strips were allowed to remain in ambient air for a period of 6 hours. The color developed matched the "X" standard. These strips were then exposed to 40 ppm of NO<sub>2</sub> for varying periods of time. The color that was developed matched the filter paper standards very well. Several additional strips were allowed to remain

exposed to ambient air for a total of 16 hours. These strips were darker blue than the "X" standard but still developed yellow color on exposure to NO<sub>2</sub>. They were judged to be about one standard darker than their actual exposure.

Several detector strips were moistened with distilled water and allowed to stand for about 2 minutes before testing. The colors developed matched the standards quite well but were somewhat more blue-green than strips that were dried in a vacuum dessicator.

An accelerated aging test was conducted by placing sealed vials of detector strips in an oven at 65 C for one week. This treatment is thought to be equivalent to aging at room temperature for one year. The strips were essentially unchanged in appearance after aging. The edges of some strips showed a faint blue color and the silica gel granules which fell off the strips and collected in the bottom of the vials were light blue. The results of the tests conducted on the aged strips are given in table IV. The results were excellent and indicate that the strips should have a shelf life greater than one year.

Several different reagent combinations were tested prior to the selection of the detector strip described above as the best choice. Strips prepared from diphenylamine in glacial acetic acid gave colors almost identical to the oxalic acid strips, but the hue changed to a more yellow color as the volatile acid was eventually lost. Diphenylamine in sulfuric acid gives a deep blue color with NO<sub>2</sub>,

TABLE IV.

COLOR DEVELOPED BY DETECTOR STRIPS AGED FOR ONE WEEK  
AT 65°C AND EXPOSED TO 40 ppm OF NO<sub>2</sub>

<i>CT Value (ppm-min)</i>	<i>Standard</i>	<i>Maerz and Paul Color Standard (Ref 8)</i>
40	A+	17F2
40	A+	17I3
40	A+	17G2
100	B	18J3
100	B	18K4
100	B	18K4
300	C	20J3
300	C	20J2
300	C	20J2

but the acid attacks the supporting material. Strips prepared from diphenylamine in acetone, ethanol, and ethyl ether gave bright yellow colors on filter paper and light green colors on the acidic surface of Eastman Chromagram Sheet. These colors were not very stable and changed rapidly to brighter yellow hues.

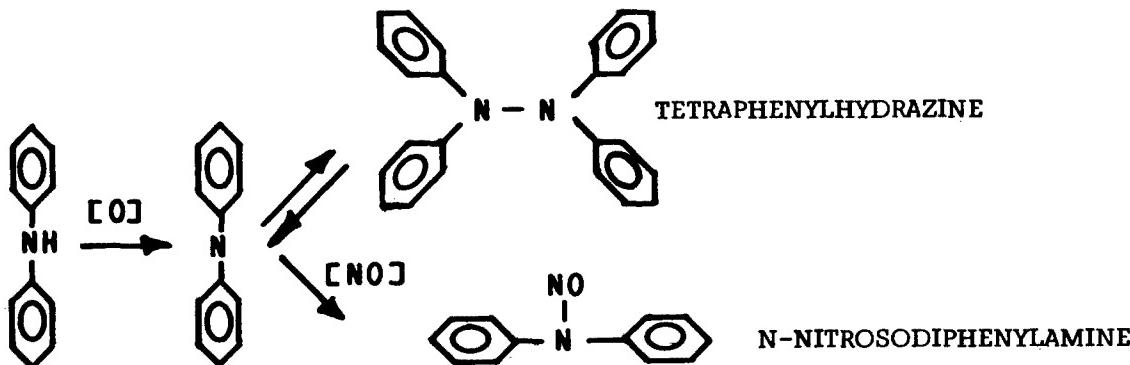
If a buffering salt such as sodium acetate is added to a methanol solution of diphenylamine, the color developed on the detector strips is yellow. The dull-yellow color initially developed changes rapidly to a brighter yellow color on standing. The buffered strips seem to be somewhat more light sensitive.

Strips were also prepared from diphenylbenzidine and o-tolidine in methanol. These strips were more sensitive to NO<sub>2</sub> than were the diphenylamine strips but were less stable on storage and aging. Both of these compounds develop blue colors with various oxidizers and perhaps will prove to be suitable for use in detecting low levels of halogenated oxidizers.

## Section VI.

### CHEMISTRY OF THE COLOR-FORMING REACTION

Fieser and Fieser discuss the formation of free radicals from diphenylamine during oxidation (ref 4). The free radicals formed are quite stable at elevated temperatures. At room temperature the diphenylamine radicals may either associate to form tetraphenylhydrazine or may react with the free radical of nitrous acid to form N-nitrosodiphenylamine as shown in Reaction I.



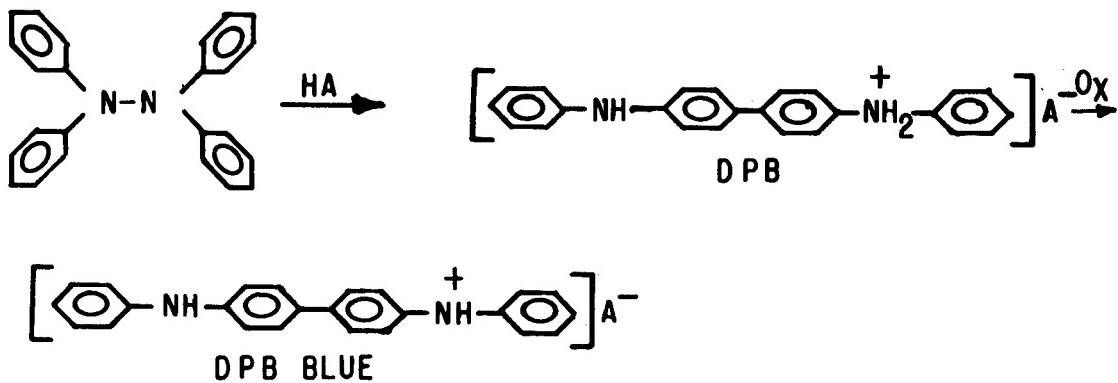
**Reaction I — The formation of tetraphenyl hydrazine and N-nitroso diphenylamine**

Schroeder discusses several mechanisms for the subsequent nitrosation and nitration of diphenylamine that may occur in smokeless powder (ref 15). The following yellow nitrated derivatives of diphenylamine are formed: 4-nitrodiphenylamine, 2-nitrodiphenylamine, 2,4'-diphenylamine, 2,2'-dinitrodiphenylamine, and various other more highly nitrated derivatives. In addition, Schroeder states that a blue pigment is formed which is probably closely related to N, N', N'-triphenyl pararosaniline.

Alexander has discussed a simple method for separating the various nitrated derivatives of diphenylamine using thin-layer chromatography (ref 1). His method was modified slightly and used to identify the pigments which are formed on exposed detector strips. A detector strip that had been exposed to  $NO_2$  was placed in a small vial and the yellow pigment extracted with 1 ml of trichloroethylene. A 50-microliter quantity of the extract was spotted on Eastman Chromagram Sheet, Type K301R2, and developed with trichloroethylene. Four distinct yellow pigments were separated and identified tentatively as 2-nitrodiphenylamine, 2,2' dinitrodiphenylamine, 2,4' dinitrodiphenylamine and 2,4 dinitrodiphenylamine. The dried chromatograph was sprayed with a solution of sodium nitrite in sulfuric acid and three additional blue spots developed which presumably are unreacted diphenylamine, N-nitrosodiphenylamine and N,N' diphenylbenzidine.

Levitsky and Norwitz have published the infrared spectra of nineteen nitration products of diphenylamine; these are invaluable for further work in this area (ref 7).

The formation of N,N' diphenylbenzidine from tetraphenylhydrazine takes place by the well known benzidine rearrangement according to Reaction II.



**Reaction II — The formation of the Acid Salt of N,N' diphenylbenzidine and diphenylbenzidine blue**

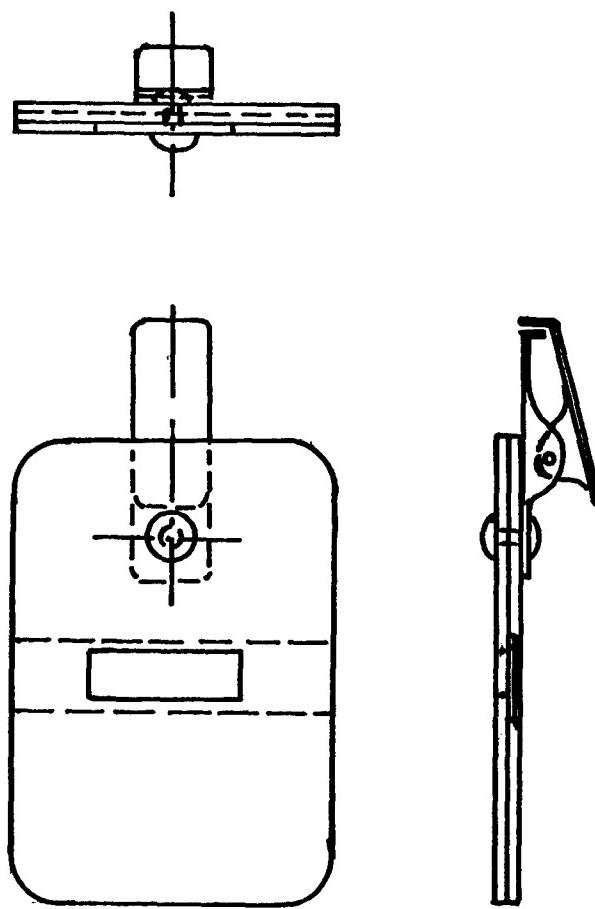
The blue pigment formed in Reaction II may be further oxidized to give other blue colored compounds. The colors observed when the detector strips were exposed to air and to NO<sub>2</sub> under various conditions can be explained quite well by the reaction scheme shown above. The oxidation of diphenylamine in air in an acid medium will tend to produce blue pigment by the association of diphenylamine free radicals and the subsequent intramolecular rearrangement and oxidation to a blue derivative. The oxidation by NO<sub>2</sub> can proceed by both direct nitrosation and nitration to give yellow nitro derivatives and by the rearrangement mechanism. The rearrangement is favored in a strong acid medium, while the dissociation of the proton from diphenylamine in basic solutions favors the nitration mechanism.

## **Section VII.**

### **PLASTIC DOSIMETER STRIP HOLDER**

A simple holder for the detector strips was fabricated from a sheet of  $\frac{1}{16}$ -inch-thick plastic. The holder is shown in figure 2. A metal clip is used to fasten the badge to the wearer's clothing. A set of color standards is fastened to the back of the badge. The badge should be made of white plastic so that slight color changes on the white detector strip can be readily observed. The detector strip is inserted in the slot at the side of the badge and viewed through the open window. The holder shown is simple, inexpensive and reusable. A somewhat more complicated holder with an ultra-violet filter was developed by the Mine Safety Appliances Company for use with their hydrazine dosimeter. By including the "X" standard to indicate an overexposure to U.V. light or other oxidizers the use of a U.V. filter was avoided.

A dosimeter manufactured by the American Gas and Chemical Company for hydrazine uses a piece of filter paper laminated between two sheets of plastic similar to the typical security badge; however, the cost of such a disposable holder is quite high when compared to a reusable holder.



**Figure 2. Plastic dosimeter badge and strip holder**

## **Section VIII.**

### **RECOMMENDATIONS**

1. The dosimeter badge described in this report should be tested in the field under conditions of actual use.
2. An independent evaluation of the performance of the strips should be made.
3. Diphenylamine, diphenylbenzidine and o-tolidine should be considered for use in a dosimeter badge for halogenated oxidizers.

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## 13. ABSTRACT

A personal colorimetric dosimeter for nitrogen dioxide has been developed. Detector strips are prepared by dipping Eastman Chromagram Sheet, Type K301R2, in a solution of diphenylamine and oxalic acid in methanol. When dry, the strips can be used to estimate the concentration-time product to which an individual has been exposed. The strips are sensitive to heat and light. Nitrogen dioxide develops a green color while other oxidizers develop a blue color. The strips are not affected by moisture and should have a shelf life greater than one year. Color standards for concentration-time products of 50, 100, and 300 ppm-minutes and a blue standard to indicate an expired strip are described. A plastic holder for the sensitive detector strips is shown.

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